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POLYURETHANE CARPET-BACKING PROCESS

This invention relates to polyurethane-backed substrates, and to a process and composition for making same.

5 Carpets and other substrates having attached polyurethane foam layers are well-known and described, for example, in U. S. Patent Nos. 3,755,212, 3,821,130, 3,862,879, 4,022,941, 4,171,395, 4,278,482, 4,286,003, 10 4,405,393, 4,483,894, 4,512,831, 4,515,646, 4,595,436, 4,611,044, 4,657,790, 4,696,849, 4,853,054, and 4,853,280. In commercial practice, these polyurethane-backed materials have been prepared from polyurethane-forming compositions based on toluene diisocyanate 15 (TDI). However, for handling reasons, it is desired to substitute a diphenylmethane diisocyanate-based material for the TDI in this application.

20 TDI-based frothed polyurethanes have an excellent balance of physical properties, including good tensile strength, load-bearing, tear strength and resiliency. Previous attempts to duplicate these properties with MDI-based systems failed to achieve this 25 excellent balance. Generally, the MDI systems yield

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inferior resiliency when formulated to provide load-bearing, tensile, tear and elongation properties equivalent to those provided by TDI-based systems. Resiliency can be improved in these systems, typically by reduction of hard segment levels (decreased use of chain extender), but with this reduction comes a loss of tensile, tear, load-bearing and elongation.

In one aspect, this invention is an improvement in a process for preparing a polyurethane-backed substrate wherein an uncured polyurethane-forming composition is applied to a surface of the substrate, gauged and cured to form an adherent backing thereto, the improvement comprising employing a polyurethane-forming composition containing

(A) an isocyanate-reactive material or mixture thereof having an average equivalent weight of 1000 to 5000,

(B) an effective amount of a blowing agent, and

(C) a polyisocyanate in an amount to provide an isocyanate index of 90 to 130, wherein at least 30 percent by weight of such polyisocyanate is an MDI soft segment prepolymer having an NCO content of 10 to 30 percent by weight.

This invention permits the use of an MDI-based isocyanate while providing for a combination of physical properties, including tensile strength, tear strength, load-bearing, elongation and resiliency, which is

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comparable to that previously achieved only with a TDI-based isocyanate.

The application of an uncured polyurethane-forming composition to a substrate and curing to form a layer of polyurethane adherent to the substrate is well-known and described, for example, in U. S. Patent Nos. 3,755,212, 3,821,130, 3,862,879, 4,022,941, 4,171,395, 4,278,482, 4,286,003, 4,405,393, 4,483,894, 4,512,831, 4,515,646, 4,595,436, 4,611,044, 4,657,790, 4,696,849, 4,853,054, and 4,853,280.

In the process, a polyurethane-forming formulation as described hereinafter is prepared, and then applied to at least one surface of a suitable substrate before it cures to a tack-free state. In one alternative embodiment, the composition is applied to the substrate before any significant curing occurs, using equipment such as a doctor knife, air knife or extruder to apply and gauge the layer. Alternatively, the composition may be formed into a layer on a moving belt or other suitable apparatus and partially cured, then married to the substrate using equipment such as a double belt (double band) laminator or a moving belt with an applied foam cushion. After application and gauging, the layer is cured, advantageously through the application of heat, such as by heating plates, a convection oven, an infrared oven, or other suitable apparatus. A cure temperature of 100°C to 170°C for a period of 1 to 120 minutes is suitable. Of course, the cure time is dependent on the temperature.

It is preferred to handle the substrate, at all times prior to final cure of the polyurethane-forming

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composition, such that mechanically induced stress is minimized. It is also preferred to cool the coated substrate to a temperature of less than about 40°C before rolling it up for storage or transportation, or cutting it into smaller segments.

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In some applications, it is desirable to coat both sides of the substrate with polyurethane, as in preparing carpet underlayment. This is easily done by coating one surface of the substrate, turning it over
10 and then coating the opposite surface. Multiple layers of polyurethane can, of course, be applied to the substrate if desired. In addition, multiple layers of substrate can be used. For example, a second substrate can be laid atop the curing polyurethane layer, so that
15 when the polyurethane has cured, a sandwich structure having an intermediate polyurethane layer is obtained. Of particular interest are carpet sandwich structures having a topmost carpet face, an intermediate
20 polyurethane foam layer, and a bottom most release layer of a nonwoven scrim, as described in U. S. Patent No. 4,853,280.

The polyurethane-forming composition of this
25 invention includes at least one isocyanate-reactive material having an equivalent weight of 1000 to 5000, an effective amount of a blowing agent, and a polyisocyanate in an amount to provide an isocyanate
index of 90 to 130, wherein at least 30 percent by
30 weight of such polyisocyanate is an MDI soft segment prepolymer having an NCO content of 10 to 30 percent by weight, said prepolymer being used in an amount to provide an isocyanate index of 90 to 130. It may

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further contain various optional components, some of which are preferably included, as discussed below.

The isocyanate-reactive material (component (A)) is an organic material or mixture thereof having an average "functionality" of 1.8 to 4 isocyanate-reactive groups per molecule and an equivalent weight per isocyanate-reactive group of 1000 to 5000. The average equivalent weight is preferably 1000 to 3000, more preferably 1300 to 2500.

The component (A) material or mixture thereof preferably has an average functionality of 1.4 to 3.0, more preferably 1.8 to 2.2, most preferably 1.9 to 2.1. The references to functionality herein refer to the actual, as opposed to nominal, functionality of the material. In the case of polyethers, especially the preferred polymers of propylene oxide, the actual functionality is usually lower than the nominal functionality, which is equal to the average number of active hydrogens/molecule of initiator compound used in its preparation. This is due to the formation of monofunctional impurities during the polymerization. The actual functionality of a polyether can be calculated from the amount of terminal unsaturation in known manner.

The isocyanate-reactive groups include hydroxyl, primary or secondary amine, thiol, carboxylic acid, imine, or other groups with an isocyanate-reactive hydrogen atom. Of these, the secondary amine, hydroxyl and imine groups are preferred due to their reactivity, and the hydroxyl groups are most preferred on the basis of cost and optimum reactivity.

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The component (A) material can be of a various composition, but preferred are polyethers and polyesters, with polyethers being especially preferred. Among the polyethers, the polymers of cyclic ethers such as C₂-C₆ alkylene oxides and tetrahydrofuran are preferred, with polymers of propylene oxide and copolymers thereof with a minor amount of ethylene oxide being more preferred. Most preferred are polymers of propylene oxide having an internal or terminal block of poly(oxyethylene) units constituting 15 to 30 weight percent of the polymer, especially those having an equivalent weight from 1900 to 2500. The preparation of such polyethers by the polymerization of the cyclic ethers in the presence of a polyhydric initiator is well-known, and described, for example, in U. S. Patent Nos. 2,902,478, 3,040,076, 3,317,508, 3,341,597 and 3,445,525.

A blowing agent is also used in the process. In this invention, the term "blowing agent" is used rather broadly to include any gas which may be incorporated into the reaction mixture, as well as other materials which volatilize, decompose, react or otherwise generate a gas under the conditions encountered in the formation of the polyurethane layer. Frothing is the preferred method of reducing density of the polyurethane, and according the preferred blowing agent is air, nitrogen, argon, helium or some other gas which is inert to the reaction mixture. Air and nitrogen are most preferred gases. Alternatively, or in conjunction with the gas, water, low-boiling organic compounds including halogenated and non-halogenated types, the so-called "azo" blowing agents can be used.

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The blowing agent is used in an amount sufficient to provide the desired density to the polyurethane. In general, the polyurethane will have a density from 1 to 50, preferably 2 to 35, more preferably 5 to 25 pounds per cubic foot when frothed with a gas. If another blowing agent is used, the density is advantageously 3 to 30, preferably 3 to 21 pounds per cubic foot.

The polyisocyanate used in this invention comprises a soft segment prepolymer of MDI, having an NCO content of 10 to 30 weight percent. This prepolymer may be used neat. However, the neat prepolymer is often a solid. Thus, it is preferred to use it at a temperature above its melting point, or as a blend with at least one other polyisocyanate, provided that it constitutes at least 30 weight percent of the polyisocyanate blend.

By "soft segment prepolymer of MDI", it is meant the reaction product of a stoichiometric excess of diphenylmethane diisocyanate (MDI) or derivative thereof and a material which has at least two isocyanate-reactive groups and a T_g below 0°C . By derivatives of MDI, it is meant three-ring or higher products derived by the phosgenation of aniline-formaldehyde condensation products (commonly known as polymeric MDI), carbodiimide-modified MDI products, commonly known as "liquid MDI", MDI which is modified with allophonate or biuret linkages which preferably have NCO contents of at least about 30 percent by weight. Pure MDI, mixtures of pure and polymeric MDI having an average functionality of no greater than about

2.2 isocyanate groups per molecule, and the liquid MDI products are preferred on the basis of cost, availability and performance.

5 The isocyanate-reactive material used in preparing the prepolymer is advantageously an organic polymer having an equivalent weight from 500 to 5000, preferably 1000 to 3000. Those materials previously described with regard to component (A) are suitable and preferred. Most preferred are hydroxyl-terminated
10 polymers of propylene oxide or block or random copolymers of a major portion of propylene oxide and a minor portion of ethylene oxide. The isocyanate-reactive materials used in preparing the prepolymer advantageously has an average functionality from 1.4 to
15 3.0, preferably 1.8 to 2.8 and more preferably 1.9 to 2.2.

The prepolymer is prepared by reacting the MDI
20 or derivative thereof and the isocyanate-reactive material at the desired proportions, typically at an elevated temperature. The proportions of components are selected so that the prepolymer has an NCO content of 10 to 30 weight percent, preferably 15 to 27 weight
25 percent. A catalyst as described hereinafter for the reaction of isocyanates and isocyanate-reactive materials may be used. The reaction is preferably conducted in the substantial absence of moisture. Suitable conditions for the preparation of MDI
30 prepolymers are described, for example, in U. S. Patent No. 4,478,960.

It is most preferred that the MDI or derivative thereof and the isocyanate-reactive material used in

preparing the prepolymer are chosen together so that the prepolymer has an average functionality of 1.9 to 2.5, preferably 1.9 to 2.4, more preferably 1.95 to 2.3 isocyanate groups per molecule.

5 In addition to the foregoing components, other optional but desirable materials may be employed. These include chain extenders, crosslinkers, fillers, surfactants, catalysts, colorants, antistatic agents, reinforcing fibers, antioxidants, preservatives and
10 water scavengers. Among these, the use of chain extenders, fillers, surfactants and catalysts are preferred.

Chain extenders which are useful in this
15 invention include compounds having an equivalent weight of up to about 500, preferably from 31 to 200, more preferably 50 to 150, and about 2 isocyanate-reactive groups per molecule. The isocyanate-reactive groups may
20 be hydroxyl, mercaptan, primary or secondary amine or imine with those containing hydroxyl groups or sterically hindered or blocked amine groups being preferred. Most preferred are hydroxyl-terminated chain
25 glycol ethers such as ethylene glycol, 1,4-butanediol, 1,6-hexamethylene glycol, dipropylene glycol, tripropylene glycol, diethylene glycol and triethylene glycol; cyclohexanedimethanol and the diverse
30 bisphenols. Suitable amine chain extenders include methylene bis(o-chloroaniline), NaCl-blocked methylene dianiline and diethyltoluenediamine.

In making backings for substrates such as textiles, a minor amount of chain extender is

advantageously used. Preferably 5 to 50, more preferably 5 to 25 parts of chain extender are used per 100 parts by weight of component (A).

Suitable catalysts include organometallic compounds and tertiary amine compounds. Suitable organometallic compounds include compounds of tin, lead, iron, bismuth and mercury. Preferred organotin catalysts include stannic carboxylates such as tin octoate and tin oleate, and stannous compounds such as dimethyltindilaurate, dibutyltindilaurate, diethyltindiacetate, di(2-ethylehexyl)tin oxide, dialkyltinbis(alkylmercaptoesters) such as di(n-butyl)tinbis(isooctylmercaptoacetate) and di(isooctyl)tin bis(isooctylmercaptoacetate). Preferred tertiary amine catalysts include trialkyl amines and heterocyclic amines, such as alkylmorpholines, 1,4-dimethylpiperazine, triethylene diamine, bis(N,N-dimethylaminoethyl)ether and N,N,N',N'-tetramethyl-1,3-butanediamine. The use of a delayed action catalyst such as an iron pentanedione or a bismuth carboxylate is often preferred, as described in U.S. Patent No. 4,611,044.

Organometallic catalysts are advantageously used in amounts ranging from 0.001 to 0.1, preferably 0.005 to 0.05 parts per 100 parts of component (A). Tertiary amine catalysts are advantageously used in amount ranging from 0.01 to 5, preferably 0.1 to 2 parts per 100 parts of component (A).

The use of a surfactant is preferred, and among the surfactants, block copolymers of ethylene oxide or mixtures of a major amount of ethylene oxide and a minor

amount of propylene oxide and dimethylsiloxane are preferred. The more preferred surfactants are block copolymers containing 15 to 40 weight percent polydimethylsiloxane, a polyether block containing at least about 60 weight percent oxyethylene units, and a molecular weight below 30,000, as described in U. S. Patent No. 4,483,894. A sufficient amount of the surfactant is used to stabilize the cells of the foaming reaction mixture until curing occurs, and to produce an even-celled foam. In general, such surfactants are used in amounts ranging from 0.01 to 2 parts per 100 parts by weight of component (A).

It is usually desirable to include a filler in the reaction mixture, in order to reduce cost and improve some physical properties. Useful fillers include materials such as calcium carbonate, aluminum trihydrate, titanium dioxide, iron oxide, barium sulfate. In frothed systems, the fillers are advantageously used in amounts ranging from 5 to 300, preferably 20 to 200, more preferably 50 to 130 parts per 100 parts component (A). Slightly higher loadings can be used in blown systems.

In preparing polyurethane-backed substrates according to the invention, the individual components of the polyurethane-forming composition are mixed and applied as a layer of preferably uniform thickness onto one surface of the substrate. It is often preferred to pre-mix all components except the polyisocyanate (and blowing agent, when the system is frothed) to form a formulated "B-side". This simplifies the metering and mixing of components at the time the polyurethane-forming composition is prepared. In preparing a

frothed polyurethane backing, it is preferred to mix all components and then blend a gas into the mixture, using equipment such as an Oakes or Firestone foamer.

5 The substrate can be a wide variety of materials to which the polyurethane layer can adhere upon curing. Plastic sheeting, cloth, paper, metal foils, felts, fiberglass scrims, woven and non-woven textiles are all suitable. Preferred are carpets, especially non-woven carpets.

10 The amount of the polyurethane-forming composition used can vary widely, from 5 to 500 or more ounces per square yard, depending on the desired characteristics of the substrate. For making carpet, 15 to 200, preferably 30 to 120 ounces of polyurethane foam are applied per square yard.

20 As described in U. S. Patent No. 4,853,054, it is often preferred to use a substantially non-cellular precoat when a textile is the substrate. Suitable precoats include latex precoats or polyurethane precoats as described in U. S. Patent No. 4,296,159 and 4,696,849. Precoat weights of 3 to 70, preferably 5 to 25 40 ounces per square yard are suitable.

30 The coated substrate made in accordance with this invention is useful for various purposes, according to the substrate selected, such as carpeting, carpet padding, draperies, tennis balls, artificial playing surfaces, wall coverings and dust abatement coverings

 The following examples are given to illustrate the invention and should not be interpreted as limiting

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it in any way. Unless stated otherwise, all parts and percentages are given by weight.

Example 1

A B-side formulation is prepared by mixing the components indicated in Table 1.

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Table 1

Component	Parts by Weight
Polyol A [®]	44
Polyol B [®]	44
Diethylene Glycol	12
Alumina Trihydrate	50
Calcium Carbonate	60
Organotin Catalyst [®]	0.014
Silicone Surfactant [®]	0.20

[®]A 2000 equivalent weight, 18% ethylene oxide-capped poly(propylene oxide) having an actual functionality of 1.70.

[®]A 2000 equivalent weight, 18% ethylene oxide-capped poly(propylene oxide) having an actual functionality of 2.30.

[®]Di(isooctyl)tin bis(isooctylmercaptylacetate).

[®]Sold as L-5440 by Union Carbide Corporation.

Sample No. 1 is prepared by mixing the components listed in Table 1 with Polyisocyanate A at a 105 index and frothing with air in an Oakes foamer to a froth density of about 420 grams/quart. Polyisocyanate A is a blend containing 50 percent by weight of a 183 equivalent weight prepolymer prepared by reacting pure 4,4'-MDI with a 2100 equivalent weight, 14 percent ethylene oxide capped poly(propylene oxide) with an actual functionality of 2.2. The remaining portion of Polyisocyanate A is a polymeric MDI having an average functionality of 2.3 and an o,p'-content of about 12 percent by weight.

The frothed mixture is then applied to the reverse side of an 18.3 ounce/yard level loop carpet having a nylon fabric tufted through a woven polypropylene primary backing. After application and

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gauging to about 1/4" thickness, the polyurethane layer is cured at 120°C for 8 minutes. For physical property testing, a portion of the mixture is gauged onto a releasable substrate, cured as above, and removed from the substrate for testing. Results of the physical property testing are as indicated in Table 2 following.

Comparative Sample A is prepared in the same manner as Sample No. 1, except for the polyisocyanate component. In Comparative Sample A, a 50/50 blend of a polymeric MDI having an average functionality of 2.3 and an o,p'-content of 12 percent by weight with a 181 equivalent weight prepolymer prepared by reacting 4,4'-MDI with a 45/55 mixture of dipropylene glycol and tripropylene glycol is used. Results of physical property testing are as reported in Table 2.

Comparative Samples B-D show how with previously known MDI-based systems, a sacrifice of tensile, load bearing and tear strength is needed to obtain acceptable resiliency. Comparative Sample B is the same as Comparative Sample A, except the calcium carbonate amount is only 47 parts. Comparative Sample C is the same as Comparative Sample B, except the diethylene glycol level is reduced to 7.7 parts and each of Polyol A and Polyol B are increased to 46.15 parts. Comparative Sample D is the same as Comparative Sample B, except the diethylene glycol level is reduced to 6.5 parts and each of Polyol A and Polyol B are increased to 46.75 parts. The results of physical property testing of each are reported in Table 2.

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Table 2

Property	Sample or Comparative Sample No.				
	1	A*	B*	C*	D*
Density, pcf	18.6	18.0	18.0	18.6	18.0
50% Compression Set, % [Ⓢ]	4.4	2.9	2.9	2.0	4.5
25%ILD, lb. [Ⓢ]	24.0	24.2	23.0	16.9	7.5
Tensile Strength, psi [Ⓢ]	62.9	85.8	65.0	44.9	23.0
Elongation, % [Ⓢ]	82	96	96	107	107
Tear Strength, pli [Ⓢ]	5.1	5.9	5.0	3.4	2.3
Resiliency, % [Ⓢ]	34	19	19	29	37

[Ⓢ]ASTM 3574-81 Test D. [Ⓢ]ASTM 3574-81 Test B. [Ⓢ]ASTM 3574-81 Test E. [Ⓢ]ASTM 3574-81 Test F. [Ⓢ]ASTM 3574-81 Test H.

Sample No. 1 is roughly comparable in
 20 properties to Comparative Sample A except for a slight decrease in tensile strength and a 50 percent improvement in resiliency. The lower tensile strength is expected, since Comparative Sample No. 1 contains a higher level of chain extender due to the use of chain
 25 extenders in the preparation of the prepolymer. Comparative Samples B, C and D show the relationship between strength properties (tensile, ILD, tear) and resiliency. These samples show that modifications which improve resiliency (reducing chain extender level) cause
 30 drastic reductions in resiliency to the point that in Comparative Sample B, unacceptable resiliency is obtained. Thus, Comparative Samples A and B, which exhibit acceptable strength properties, obtain such only at the cost of poor resiliency.

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CLAIMS:

1. In a process for preparing a polyurethane-backed substrate wherein an uncured polyurethane-forming composition is applied to a surface of the substrate, gauged and cured to form an adherent backing thereto, the improvement comprising employing a polyurethane-forming composition containing

(A) an isocyanate-reactive material or mixture thereof having an average equivalent weight of 1000 to 5000,

(B) an effective amount of a blowing agent, and

(C) a polyisocyanate in an amount to provide an isocyanate index of 90 to 130, wherein at least 30 percent by weight of such polyisocyanate is an MDI soft segment prepolymer having an NCO content of 10 to 30 percent by weight.

2. The process of Claim 1 wherein said soft segment prepolymer is the reaction product of a stoichiometric excess of MDI, polymeric MDI or liquid MDI and an organic polymer having an equivalent weight

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from 1000 to 3000 and an average functionality from 1.4 to 3.0.

5 3. The process of Claim 2 wherein said organic polymer is a hydroxyl-terminated polymer or propylene oxide or a block or random copolymer of a major portion of propylene oxide and a minor portion of ethylene oxide.

10 4. The process of Claim 3 wherein component (A) is a polymer of propylene oxide or a copolymer of propylene oxide and a minor amount of ethylene oxide, having a functionality of 1.8 to 2.2.

15 5. The process of Claim 3 wherein said polyurethane-forming composition further contains a silicone surfactant and a chain extender.

20 6. The process of Claim 5 wherein the substrate is a textile.

25 7. The process of Claim 6 wherein a release layer is applied to the surface of the polyurethane-forming layer prior to the curing thereof.

30

INTERNATIONAL SEARCH REPORT

International Application No. **PCT/US91/09481**

I. CLASSIFICATION OF SUBJECT-MATTER (If several classification symbols apply, indicate all) ⁶ According to International Patent Classification (IPC) or to both National Classification and IPC: U.S. CL: 427/244, 358, 272, 289.9, 412 IPC(5): B05D 5/00, 3/12, 3/02, 7/00		
II. FIELDS SEARCHED Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
U. S. CL:	427/244, 373, 389.9, 412, 358, 172	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹		
Category ¹	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹⁰	Relevant to Claim No. ¹²
X	US, A, 4,913,958, SKAGGS ET. AL., 03 APRIL 1990 (Col. 2, lines 35-55, Cols. 3-5).	1-6
X	US, A, 3,849,156, MARLIN ET. AL., 19 NOVEMBER 1974 (Col. 4, Col. 8, lines 1-26, Col. 9, line 59-Col. 10, line 26, Col. 14, lines 27-64, Col. 16, lines 55-68).	1,2,3,4
A	US, A, 4,657,790, WING ET. AL., 14 APRIL 1987 (Col. 6, lines 51-60).	7
A	US, A, 4,035,529, MEISERT ET. AL., 12 JULY 1977 See entire document.	1, 2
<p>¹³ Special categories of cited documents: ¹⁴</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubt on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"A" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search 09 MARCH 1992		Date of Mailing of this International Search Report 08 APR 1992
International Searching Authority ISA/US		Signature of Authorized Officer <i>Andie Robinson</i> DIANA DUDASH

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(54) Title: POLYURETHANE CARPET-BACKING PROCESS (57) Abstract Polyurethane-backed substrates such as attached cushion carpeting are prepared from a polyurethane forming composition based on a soft segment prepolymer of MDI or an MDI derivative. The use of the prepolymer provides a backing having good strength properties and good resiliency.		

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